EP 22497 (3)

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M3 - [01] J0 J012 J3 J372 M280 M311 M312 M313 M314 M315 M316 M321 M331 M332 M333 M342 M382 M391 M416 M620 M782 M903 Q133 R043
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PA - (ASAH) ASAHI CHEM IND CO LTD
PN - JP61188458 A 19860822 DW198640 005pp
- JP6055886B B2 19940727 DW199428 C08L77/06 004pp
PR - JP19850026387 19850215
XA - C1986-112937
XIC - C08K-005/09 ; C08K-005/10 ; C08K-005/20 ; C08L-071/02 ; C08L-077/06 ; (C08L-071/02 C08L-077/06)
AB - J61188458 Nylon 46 moulding resin compsn. contg. (1) nylon 46 100 pts. wt., and lubricant selected from (2)-(5), 0.01-1.0 pts.wt., (2) bisamide type cpd., (3) Gp= I,II,III metal salt of higher fatty acid (4) polyethylene glycol, (5) 26-32C aliphatic carbonic acid (deriv.).
- USE/ADVANTAGE - Nylon-46 has high melting point and strong mechanical properties, but poor mouldability, and, it is hard to mould into complicated shaped products or thin shaped products, by usual injection moulding method. It also has defect of mol. wt. decreasing at high temp., however this moulding compsn. has good mouldability, good chemical stability, heat resistance. Additive lubricant is effective in improving these properties. (5pp Dwg.No.0/1)
AW - GROUP-I GROUP-II GROUP-III
AKW - GROUP-I GROUP-II GROUP-III
C - C08L77/06 C08L71/02
IW - NYLON MOULD RESIN COMPOSITION HIGH MELT POINT CONTAIN NYLON LUBRICATE DI AMIDE METAL HIGH FATTY ACID SALT POLYETHYLENE GLYCOL ALIPHATIC CARBONIC ACID DERIVATIVE
IKW - NYLON MOULD RESIN COMPOSITION HIGH MELT POINT CONTAIN NYLON LUBRICATE DI AMIDE METAL HIGH FATTY ACID SALT POLYETHYLENE GLYCOL ALIPHATIC CARBONIC ACID DERIVATIVE
NC - 001
OPD - 1985-02-15
ORD - 1986-08-22
PAW - (ASAH) ASAHI CHEM IND CO LTD
TI - Nylon-46 moulding resin compsn. with high melting point - contg. nylon-46, lubricant e.g. bis-amide, metal higher fatty acid salt, polyethylene glycol and aliphatic carbonic acid deriv. etc.

EP 22497 (1)

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(72) Inventors; and
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(54) Title: ADHESIVE

(57) Abstract

Inert polymeric materials, for example polyamides, are bonded by applying to one or both surfaces a composition comprising a compound having a phenolic group and having substituents which reduce the toxicity of the compound, the composition being a solvent for the inert polymer, and mating the surfaces. Preferred compounds are carvacrol, and/or thymol. A eutectic mixture of carvacrol and thymol may be used to -20°C. Adhesives according to the invention have a polyamide dissolved in the composition.

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Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

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DE	Germany, Federal Republic of	MC	Monaco	US	United States of America
DK	Denmark				

Title: ADHESIVE

FIELD OF THE INVENTION

The present invention relates to a method of bonding inert polymers and to adhesive compositions for use in the method.

BACKGROUND OF THE INVENTION

Inert polymeric materials, such as polyamide compositions, have been bonded in the past by application to the surface of the materials of a solvent effective for the materials to be joined, resulting in interpenetration in the bonding zone of the polymers from both surfaces. Such adhesion is due primarily to cohesion within the solid polymer, thus filling the bonding region after the solvent evaporates. Alternatively, where the degree of mating of the two surfaces to be bonded is too great for the above mechanism to give the desired adhesive strength, an

adhesive can be prepared containing pre-dissolved polyamide so to as enhance the "gap filling" ability of the adhesive.

Materials currently in use as solvents of the above mentioned kind are few due to the inertness of the polymer to solvent attack. The solvents currently used generally are good hydrogen bonders, contain some hydrophobic moieties and often acidic hydrogen. Such a combination of properties results in the known solvents being toxic to man. The toxicity is often systemic and via external exposure, with risk of burns and lesions. The most well-known class of such solvents are phenols which are highly toxic to man. The most common known solvents for polyamide are cresols and chlorophenols both of which are known to be harmful substances. Other solvents include various fluoroalcohols which are generally highly toxic to man.

The main objects of the present invention are to provide an adhesive suitable for use in bonding inert polymeric materials, especially polyamides, and having low toxicity, particularly low systemic toxicity; providing high adhesion strength, particularly with reference to tensile and peel testing; providing rapidity of curing such that joint strength is acquired quickly; having broad temperature applicability; and having desirable gap filling capability. Desirably the adhesive should be suitable for joining pipe systems

to be pressurized, e.g. gas pipes and fittings and should have a good shelf life.

An adhesive meeting the above objects would have applications in the food/water processing areas for which currently available (relatively toxic) adhesives are unsuitable.

DISCLOSURE OF THE INVENTION

According to one aspect, the invention consists in a method for adhering one inert polymeric material surface to another comprising the steps of:

(i) applying to at least one of the surfaces a substantially non-toxic composition comprising at least one compound having a phenolic hydroxyl group and one or a combination of substituents which reduce the toxicity of the compound, said composition having the capability to act as a solvent for said inert polymeric material, and

(ii) bringing said surfaces into bonding relationship.

For preference, the composition comprises a dialkyl phenol. Carvacrol and/or thymol, or more preferably an eutectic mixture of carvacrol and thymol, have been found to be especially suitable for bonding polyamides.

According to a second aspect, the invention consists in an adhesive for inert polymeric materials comprising at least one compound having a phenolic hydroxyl group and one or a combination of substituents which reduce the toxicity of the compound, the

composition having the capability to act as a solvent for said inert polymeric material.

PREFERRED EMBODIMENTS OF THE INVENTION

The invention will now be more particularly described with reference to various embodiments by way of example only. The invention will be exemplified with particular reference to the bonding of polyamides.

Polyamides are polymers in which regularly occuring amide groups are part of a methylene polymer chain as shown in Figure 1.

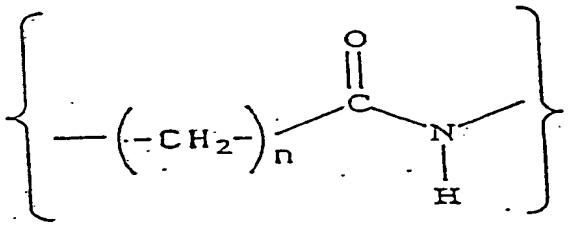


Figure 1

Specific polyamides (abbreviated to "PA") are identified by numerals which indicate the number of carbon atoms in the carbon chain of each repeating unit of the polymer. Thus, with reference to Figure 1, PA 11 is the name given to the polyamide polymer in which n = 10, and PA 12 is the name given to the polyamide polymer in which n = 11 and so on. In addition, AABB polyamide polymers are known and in that case a designation such as PA 12/12 indicates that the chain is derived from diamine units having 12 carbon atoms and dicarboxylic

acid units having 12 carbon atoms. PA 6/12 indicates a chain derived from 6-carbon-atom diamine units co-polymerized with 12-carbon-atom dicarboxylic acid units and so on.

In an attempt to develop an improved adhesive for polyamides a large number of compounds were screened as solvents for PA 11.

The solvents were tested alone and in mixtures and were selected from:

Aromatic and aliphatic amines

Aromatic and aliphatic alcohols

Aromatic and aliphatic halides

Aromatic and cyclic aliphatic ethers

Aromatic and aliphatic acids

Metal salts in combination with various organic solvents

Of the compounds tested from the above categories none were effective solvents for PA 11.

Phenols tested were found to be effective as solvents. However, these were initially rejected as being as, or more, toxic than methyl phenol (cresol), and because most have melting points above room temperature. Some high melting point phenols, when dissolved in a halogenated solvent, were found to form a reasonably effective glue but those compositions had little capacity to dissolve PA 11 to give gap filling abilities. Moreover, either the phenol or solvent or

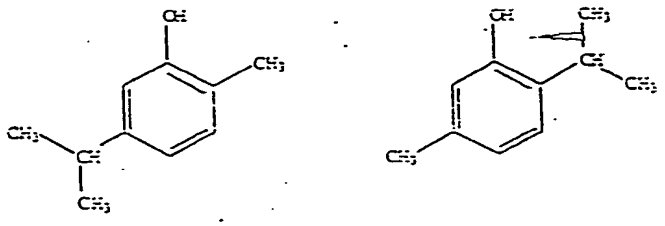
both were harmful or environmentally undesirable.

According to the present invention, there is applied to a surface of the polyamide to be bonded a compound having a phenolic hydroxyl group and one or a combination of substituents which reduce the toxicity of the compound, the composition having the capability to act as a solvent for the polyamide. For preference, the compound is selected from dialkyl phenols, the substituents being selected from the group consisting of methyl, ethyl, propyl, butyl, iso-propyl, sec-butyl and tert-butyl.

A highly preferred compound for application as a polyamide adhesive is carvacrol (2-METHYL, 5-ISOPROPYL PHENOL) which has a single phenolic hydroxyl group having a meta-isopropyl substituent and an adjacent methyl substituent.

Another highly preferred compound for use in the invention is thymol (5-METHYL, 2-ISOPROPYL PHENOL) which has a single phenolic hydroxyl group sterically hindered by the ortho-isopropyl substituent and a meta methyl substituent.

The chemical structures of carvacrol and thymol are shown below:



Carvacrol ("Isothymol") Thymol

Figure 2

The isopropyl and methyl substituents together reduce the toxicity of the compound, that is to say the substituted compound is less toxic than the corresponding unsubstituted phenol.

It has been found that carvacrol and thymol are particularly effective solvents for polyamides.

Carvacrol has a melting point of about 1°C and is therefore liquid at room temperature, whereas thymol, having a melting point of about 50°C, is a solid under ambient conditions.

Both compounds occur naturally in essential oils commonly used as food flavorants; carvacrol is the major constituent of oil of origanum, and thymol the major constituent of oil of thyme. Both are manufactured synthetically for a variety of commercial uses which include perfumery, antibacterial applications such as toothpastes, mouth washes, and flavour additives for food, for which both compounds have been approved by the Food and Drug Administration of the USA. Toxicity data currently available gives both compounds the relatively innocuous rating of "irritating". Both compounds are thus substantially harmless in comparison with, for example, cresols or chlorphenols. However, grades of sufficient purity should be employed.

In particular, it is preferred to use a combination of carvacrol and thymol, desirably mixed in the proportion 2:1 by weight.

Carvacrol itself is a satisfactory solvent for various inert polymeric materials, particularly polyamides, and provides acceptable adhesive bond strength. However, it is unusable at below about 1°C as it solidifies.

Thymol has a melting point of about 50°C, and although it gives a satisfactory adhesive strength when used above this temperature, it is often inconvenient for use on its own due to its high melting point.

However, when carvacrol and thymol are mixed in the weight proportion 2:1 they form an eutectic mixture which remains liquid to below -20°C. The eutectic mixture is therefore advantageously chosen as the optimum solvent base for use in the present invention.

In use, the mixture is applied to one, or preferably to both, surfaces of polyamide parts to be bonded and the treated surfaces are then mated face to face, that is to say are brought into bonding relationship.

Although this solvent base alone can form a satisfactory adhesive bond between well-mating polyamide surfaces, it has little or no gap-filling properties and is of too low a viscosity to be applied effectively to inclined surfaces. To overcome these deficiencies the adhesive is preferably formulated to contain up to 10% of a polyamide in solution in the carvacrol/thymol solvent base. The concentration and type of dissolved

polyamide in the adhesive formulation is chosen to suit the type of service application desired. PA 11 is the preferred dissolved polymer for joining a PA 11 surface to either a PA 11 or a PA 12 surface, while PA 12 is preferred for bonding two PA 12 surfaces. The polyamide or polyamides are preferably added to the solvent as a powder.

At any given temperature, increasing the level of dissolved polyamide produces an adhesive of increased viscosity, which has improved gap-filling properties, but a lengthened set time. Conversely a lower polyamide concentration lowers the viscosity of the adhesive, reducing the gap-filling properties, but speeding up the set time. Furthermore, temperature strongly affects the adhesive's properties; both viscosity and set time increase with decreasing temperature. The adhesive may therefore be formulated with varying dissolved polyamide concentrations suited to the intended temperature of use, which maintain optimum set time and viscosity. Low ambient temperatures require a lower level of dissolved polyamide and, conversely, high ambient temperatures require a higher level.

In particular, for an adhesive to be used to join polyamide pipe and fittings conforming to Australian Standard 2944 at ambient temperatures between 10°C and 30°C the preferred concentration of dissolved polyamide is 5% by weight.

Although AS 2944 relates to gas pipes, the invention is applicable to pipes used for other purposes and for joining tubes.

The adhesives of the present invention are preferably used for adhering PA 11 or PA 12 surfaces together by a solvent adhesive mechanism. This mechanism may be explained in simple terms as follows:

1. The adhesive dissolves each of the two surfaces to be bonded resulting in interpenetration in the bonding zone of polymer from both surfaces as a solution in the adhesive;

2. The solvent is lost by evaporation or penetration and diffusion into the body of the substrate and/or eventual evaporation, leaving the bonding zone comprised of solid polymer. The resultant adhesion between the two substrates is due primarily to cohesion within the solid polymer that fills the bonding region after loss of solvent. Where the degree of mating of the two surfaces is insufficient for the above mechanism to give the desired adhesive strength, the adhesive may be prepared to contain pre-dissolved polyamide so as to enhance the gap-filling ability of the adhesive.

Preferred embodiments of this invention have the advantages of:

1. Being relatively non-toxic in comparison with those previously used and having no systemic toxicity;

2. Having an unlimited shelf life and remaining liquid

down to a temperature of -20°C;

3. Having viscosities suitable for applying a film of the minimum thickness necessary to form an adhesive bond over the temperature range -20°C to +35°C;

4. Forming effective adhesive bonds when applied at ambient temperatures within the range -20°C to +35°C;

5. Setting within 30 minutes of the assembly of a joint between a polyamide pipe and corresponding pipe fitting conforming to Australian Standard 2944, the joint being able to withstand an internal pressure test of 320 kPa, and a tensile load equivalent to 60% of the yield load of the pipe after this period;

6. Forming adhesively bonded joints as per 5 above which attain maximum strength within a week and which will withstand a tensile load in excess of the yield load of the pipe.

Adhesives prepared in accordance with the present invention may be varied extensively with regard to setting rate, and hence may be used in service applications where rapid setting is required, as well as those where rapid setting may be disadvantageous.

The adhesives are not limited to use with PA 11 and PA 12 and are also applicable to PA 12/12, PA 10/10, PA 6/12, PA 6/10, PA 6/6, PA 6, PA 4/6 and other polyamides.

Although exemplified by reference to bonding of polyamides it is anticipated that adhesives according to

the invention may be used to bond other materials for example polyamide block copolymers, branch chain polyamides, etc. and the suitability for bonding particular materials can be determined by routine testing having regard to the teaching hereof.

Although thymol and carvacrol have been found to be particularly effective in combination each can be used alone or with other compounds.

Although specific embodiments of the present invention have been described in detail herein, it should be understood that the present invention is not restricted to the preferred embodiments.

As will be apparent to those skilled in the art from the teaching hereof, other substituted phenols in which the substituents are effective to reduce the toxicity of the compound, and which are effective in the dissolution of polyamides may be selected for use in adhesives according to the invention.

Dialkyl phenols in which the substituents are selected from the group methyl, ethyl, propyl, butyl, isopropyl, sec-butyl and tert-butyl are, or are likely to be, suitable for use in an adhesive for polyamides as herein disclosed, and can be screened for toxicity and solvation efficiency by known methods. Combinations of such compounds which are liquid at ambient temperature are especially preferred but it is sufficient that the composition be liquid at the temperature of application to the polyamide.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A method for adhering one inert polymeric material surface to another comprising the steps of:

(i) applying to at least one of the surfaces a composition comprising at least one compound having a phenolic hydroxyl group and one or a combination of substituents which reduce the toxicity of the compound, said composition having the capability to act as a solvent for said inert polymeric material, and

(ii) bringing said surfaces into bonding relationship.

2. A method according to Claim 1 wherein the inert polymeric material is a polyamide.

3. A method according to Claim 1 or Claim 2 wherein the at least one compound is a dialkyl substituted phenol the substituent being selected from the group consisting of methyl, ethyl, propyl, butyl, iso-propyl, sec-butyl, and tert-butyl alkyl substituents.

4. A method according to any one of the preceding claims wherein the compound has an isopropyl substituent.

5. A method according to any one of the preceding claims wherein the compound is carvacrol.

6. A method according to any one of claims 1 to 4 wherein the compound is thymol.

7. A method according to any one of claims 1 to 4 wherein the composition comprises a mixture of carvacrol and thymol.

8. A method according to Claim 7 wherein the

composition comprises a mixture of carvacrol and thymol in proportions at or near those of an eutectic mixture.

9. A method according to any one of the preceding claims wherein the at least one inert polymeric material surface to be bonded is a polyamide selected from the group comprising PA 11, PA 12, PA 12/12, PA 6/12, PA 10/10, PA 6/10, PA 6/6, PA 6 and PA 4/6.

10. A method according to any one of the preceding claims wherein the composition further comprises a dissolved polyamide.

11. A method according to Claim 10 wherein a polyamide selected from the group consisting of PA 11, PA 12, PA 10/10, PA 12/12, PA 6/12, PA 6/10, PA 6/6, PA 6 and PA 4/6 is dissolved in the composition prior to applying to the inert polymeric material.

12. A method according to Claim 10 wherein the selected polyamide is PA 11 or PA 12.

13. A method substantially as herein described with reference to any one of the examples.

14. An adhesive for inert polymeric materials comprising at least one compound having a phenolic hydroxyl group and having one or a combination of substituents which reduce the toxicity of the compound, the composition having the capability to ~~act~~ as a solvent for said inert polymeric material.

15. An adhesive according to Claim 14 comprising a dialkyl substituted phenol, the substituent being

selected from the group consisting of methyl, ethyl, propyl, butyl, iso-propyl, s c-butyl and tert-butyl alkyl substituents.

16. An adhesive according to claims 14 or 15 further comprising a dissolved polyamide.

17. An adhesive according to any one of claims 14 to 16 wherein the compound has an isopropyl substituent.

18. An adhesive according to any one of claims 14 to 16 wherein the compound is carvacrol.

19. An adhesive according to any one of claims 14 to 17 wherein the compound is thymol.

20. An adhesive according to claims 14 to 17 wherein the composition comprises a mixture of carvacrol and thymol.

21. An adhesive according to Claim 20, wherein the proportion of carvacrol to thymol is, or is close to, that of an eutectic mixture.

22. An adhesive according to any one of claims 14 to 20 wherein the dissolved polyamide comprises a polyamide selected from the group comprising PA 11, PA 12, PA 10/10, PA 12/12, PA 6/12, PA 6/10, PA 6/6, PA 6 and PA 4/6.

23. An adhesive according to Claim 20, wherein the dissolved polyamide is PA 11 or PA 12.

24. An adhesive substantially as herein described with reference to any one of the examples.

25. An adhesive according to any one of claims 13 to 21

when applied to a polyamide surface.

26. A polyamide product bonded with another polyamide product by means of an adhesive according to any one of claims 13 to 25.

27. A polyamide pipe bonded with another pipe or pipe fitting by means of an adhesive according to any one of claims 13 to 21.

INTERNATIONAL SEARCH REPORT

International Application No. PCT/AU 89/00496

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) 6

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[4] C08J 5/12, C09J 3/00, 3/14, C08L 77/02, 77/06

II. FIELDS SEARCHED

Minimum Documentation Searched 7

Classification System	Classification Symbols
IPC	C08J 5/12, C09J 3/00, 3/14, C08L 77/02, 77/06

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched 8

III. DOCUMENTS CONSIDERED TO BE RELEVANT 9

Category*	Citation of Document, 11 with indication, where appropriate, of the relevant passages 12	Relevant to Claim No 13
X	'The Merck Index' 10th Edition, published 1983 by MERCK & CO, INC (USA) page 261-262 No 1855 Carvacrol	(17-18)
X	'The Merck Index' 10th Edition, published 1983 by MERCK & CO, INC (USA) page 1347 No 9246 Thymol	(17, 19)
X	US,A, 1946057 (BRITTON) 6 February 1934 (06.02.34)	(1-14)
X	DE,A, 953996 (ATLAS POWDER COMPANY) 7 June 1956 (07.06.56)	(1-4, 13, 17, 24)
X	CH,A, 367324 (GENERAL ANILINE & CILM CORPORATION) 29 March 1963 (29.03.63)	(1-4, 13, 17, 24)

continued

* Special categories of cited documents: 10

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
15 February 1990 (15.02.90)	22/02/90
International Searching Authority	Signature of Authorized Officer
Australian Patent Office	G. MASTERS Gordon Masters

Form PCT/ISA/210 (second sheet) (January 1985)

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

A	US,A, 3673145 (MINAMI et al) 27 June 1972 (27.06.72)	
A	EP,A2, 0311292 (KLIMENT OHRIDSKI) 12 April 1989 (12.04.89)	

V. [] OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE 1

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1.[] Claim numbers ..., because they relate to subject matter not required to be searched by this Authority, namely:

2.[] Claim numbers ..., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3.[] Claim numbers ..., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4 (a):

VI. [] OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING 2

This International Searching Authority found multiple inventions in this international application as follows:

1.[] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2.[] As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3.[] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4.[] As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

[] The additional search fees were accompanied by applicant's protest.

[] No protest accompanied the payment of additional search fees.

CORRECTED VERSION*

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT

(51) International Patent Classification 4 :
C08J 5/12, C09J 3/00, 3/14
C08L 77/02, 77/06

A1

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PJ 1536 18 November 1988 (18.11.88) AU

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(57) Abstract

Inert polymeric materials, for example polyamides, are bonded by applying to one or both surfaces a composition comp ing a compound having a phenolic group and having substituents which reduce the toxicity of the compound, the composition ing a solvent for the inert polymer, and mating the surfaces. Preferred compounds are carvacrol, and/or thymol. A eutectic m ture of carvacrol and thymol may be used to -20°C. Adhesives according to the invention have a polyamide dissolved in composition.

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